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Promising Properties and System Demonstration of an Environmentally Benign Yellow Smoke Formulation for Hand-Held Signals

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ABSTRACT: A novel yellow smoke formulation, based on the environmentally benign dye Solvent Yellow 33, has been validated as a replacement for the current formulation specified for the M194 yellow smoke hand-held signal. Sieve testing has revealed a fine granulation for the candidate mixture, centered at 53 μm. In addition, compressive strain testing has shown that the mechanical (crush) strength of pellets derived from the candidate mixture was 391 kg. Both of these properties validate the viability of the candidate composition during manufacturing and deployment. Extensive static burn testing in full-sized prototype signal assembly hardware provides insight into new ignition train concepts. The full sized prototype assembly has also been demonstrated by flight tests in actual system hardware containing the rocket motor and associated propellant.

KEYWORDS: *Pyrotechnics, Formulation, System engineering, Solvent Yellow 33, Colored smoke, Product lifecycle management*

INTRODUCTION

In recent years, there has been a growing trend toward making military pyrotechnics more sustainable throughout the entire armament lifecycle.[1−3] Recent efforts in our laboratory have entailed the replacement of chemical ingredients that pose risks to different phases of the product lifecycle such as supply chain, manufacturing, and demilitarization.[4−14] Recently, we reported on the development of a new formulation, based on Solvent Yellow 33 instead of the toxic yellow dyes formerly specified, intended for incorporation into the U.S. Army's M194 yellow smoke hand-held signal (HHS).[15−17] Table 1 shows a modified version of this previously reported composition, one without fumed silica hereafter referred to as formulation A. This formulation consists of Solvent Yellow 33 (colored smoke dye), sucrose (fuel), potassium chlorate (oxidizer), stearic acid (lubricant, processing aid), and hydromagnesite ($Mg_5(CO_3)_4(OH)_2{\cdot}4H_2O$, endothermic coolant).

Table 1. Chemical Makeup of Formulation A

ingredients	wt %
Solvent Yellow 33	37.5
$KClO_3$	34.5
sucrose	21.5
$Mg_5(CO_3)_4(OH)_2{\cdot}4H_2O$	5.5
stearic acid	1.0

It is particularly noteworthy that this formulation does not specify a discrete binder and is composed of all solid ingredients. Thus, blending of formulation **A** can be achieved by simply combining all of the solid ingredients into a container and tumbling end-over-end, with no need for any organic solvents that may pose an additional environmental hazard. Then, the resulting formulation can be consolidated at high pressure into a cardboard tube, affixed with a parachute, and finally loaded into hand-held signal hardware capable of sending the assembly approximately 800 feet in the air. At this apex, the yellow smoke candle begins to burn, producing a highly visible stream of yellow smoke for 9−18 s.[15−17]

Although formulation **A** has been demonstrated statically with acceptable results, some additional testing of the candidate replacement formulation was necessary to assess the viability of our dry processing method before transitioning to production and subsequently testing in actual system hardware. Our initial concern was that formulation **A**, with a lack of a discrete binder, would not be amenable to manufacturing due to dust formation. In addition, candles derived from formulation **A** may not have sufficient mechanical strength to sustain the force exerted upon launch from the rocket motor. Thus, additional information was needed regarding such physical properties as granulation and mechanical strength. Accordingly, we report here on the next stage of development efforts for formulation **A**, including measurement of promising physical properties as well as outdoor performance testing results in actual system hardware.

RESULTS AND DISCUSSION

Sieve Analysis. The granulation of a novel pyrotechnic formulation is always a useful property to evaluate in order to gain some insight into the propensity for it to generate dust. This property becomes especially important when transitioning

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14. ABSTRACT
A novel yellow smoke formulation, based on the environmentally benign dye Solvent Yellow 33, has been validated as a replacement for the current formulation specified for the M194 yellow smoke hand-held signal.

15. SUBJECT TERMS

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a formulation from the pilot plant to manufacturing scale, the latter typically performed at about 54.5 kg. Accordingly, formulation **A** was added to a vertical stack of sieves, arranged from top to bottom in descending order of pore size, which was then mechanically agitated. The fraction retained on each sieve was weighed, and the percentage corresponding to each fraction is reported below in Table 2. Note especially that the mass recovered for formulation **A** is within 1% error.

Table 2. Granulation Data for Formulation A

U.S. standard sieve no.	percent retained
40	0.16
70	1.11
140	11.01
270	61.07
325	19.23
pan	6.63
total	99.21

As shown in Table 2, the mass of formulation **A** was distributed in a bell-shaped fashion centered at the 270 mesh sieve, corresponding to a pore size of 53 μm.[18] This value reflects the average particle size of the major individual components of the mixture: sugar, $KClO_3$, and Solvent Yellow 33 (see Experimental Section). This means that the processing of formulation **A** does not appear to introduce any self-agglomeration among the granules even in the presence of the sugar (sucrose) and waxy stearic acid additive. Also, we observed a lack of significant dust formed during any of the three 100 g trials with the mechanically agitated sieve stack. In summary, the granulation properties of formulation **A** seemed acceptable to advance to production.

Compressive Testing of Miniature Pellets. The next step to validate formulation **A** was to assess its mechanical integrity when packed into the hand-held signal form factor. This is important to ensure the full-sized candles can sustain the impulse subjected to the payload when launched from the signal rocket motor. Accordingly, small pellets derived from formulation **A** were pressed into a cylindrical geometry and subjected to compressive testing. The crush behavior exhibited by formulation **A** was uniform between all pellets tested, and a representative trace is presented below in Figure 1. Notice how the load steadily increases after the pressure transducer makes initial contact with the pellet until the sample becomes plastic and can no longer sustain an applied load.

The average results of compressive testing of formulation **A** are summarized in Table 3. Despite the lack of a formal binder, small 3 g pellets derived from formulation **A** proved quite

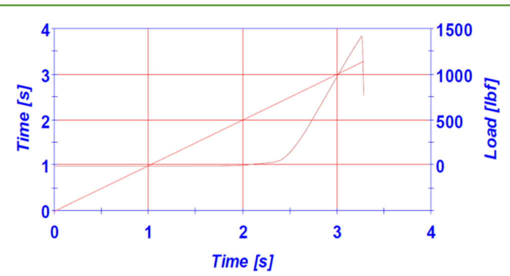


Figure 1. Representative crush trace of a pellet derived from formulation **A**. The right *y*-axis corresponds to a load range from 0−680.4 kg.

Table 3. Compressive Stress Test Data for Formulation A

formulation	load at break (kg)	compressive stress at break (kPa)
A	391.0	30 268

robust, breaking at a force of 391 kgf. While the impulse from the actual hand-held signal expelling charge cannot be easily calculated for comparison, this crush strength test provides a figure of merit for the ruggedness of candles derived from formulation **A**.

Outdoor Static Testing. After having assessed the mechanical properties of formulation **A**, the next step was to conduct static burn testing in the full-up signal assembly hardware. Although we have previously reported laboratory testing in the cardboard tube format,[15] the current aim was to include the protective discs and the parachute anchor roll-crimped into the bottom of the candle. This was necessary to ensure that the specified signal assembly hardware will remain intact during the ignition and combustion propagation phases of burning as they would occur during flight.

To this end, formulation **A** was consolidated into a slightly modified version of the currently specified signal assembly format, depicted as the baseline configuration in Figure 2. This

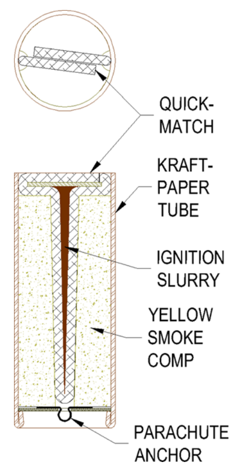


Figure 2. Cross-sectional depiction of the baseline configuration with quickmatch arms (hashed) wrapped around protector disc (buff-colored).

format stipulates folding a 27.94 cm strip of quickmatch in half, insertion of the folded end into the central candle bore flush with a fiberboard protector disc, and lacing both ends of the quickmatch around another fiberboard protector disc at the ignition end of the candle. Also, a thermate-based ignition slurry was filled into the inner core of the candle and also applied to the top face of the protector disc at the ignition end of the candle to make contact with the folded quickmatch ends. There are only two key differences between the current specification and this new baseline configuration: (1) The tube is now composed of cardboard instead of stainless steel. (2) The quickmatch strand is no longer laced through the plug end protector disc. Both of these modifications were made to reduce lifecycle costs and, particularly for the first modification, to minimize collateral damage that could be inflicted if the tube were reclaimed and repurposed by an enemy.

After consolidating formulation **A** into this configuration, 10 candles were conditioned hot (71 °C), another 10 cold (−54 °C), and 10 more at ambient temperature (~21 °C) overnight.

The three sample sets were tested statically the following day, and these results are summarized below in Table 4. Not surprisingly, our previous laboratory results at ambient temperature were validated as each assembly burned within the required time range of 9−18 s. Similarly for the heat-treated samples, no deviation from the 15 s burn time was observed, and a steady fountain of yellow smoke was produced with sufficient color quality. In the cold-treated samples, however, there was a substantial extension of the burn time to 17.79 s. This burn time extension at low temperature is common for many pyrotechnic items and is caused by the greater difference between the combustion temperature of the energetic material and the temperature at which the sample is conditioned. In other words, at lower initial temperature it takes longer to heat material ahead of the burning front to an appropriate combustion temperature.[19]

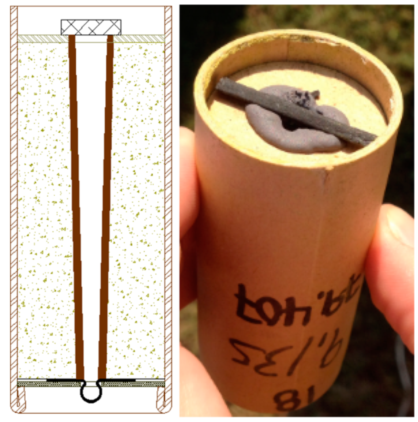
Table 4. Variable Temperature Performance Testing of Formulation A in Baseline Configuration

conditioning temperature range	burn time (s)	plug failure rate (%)
ambient	15.52	40
hot	15.74	40
cold	17.79	20

Despite the versatility exhibited by formulation **A** across three different temperature ranges, a systemic problem arose during the ignition process in many of the tested candles in each range—ejection of the bottom parachute anchor from the signal assembly (Figure 3). In every instance, ejection occurred immediately after the ignition event, just before the propagation front migrated to the yellow smoke candle. Presumably, the ejection was caused by the voluminous gases produced by an excessive amount of ignition complex (quickmatch + igniter slurry) loaded within the central bore of the candle. This hypothesis is validated by the fact that the plug failure rate drops from 40% at hot and ambient temperatures down to only 20% at the cold temperature. Clearly, the reduced rate of gas production at cold temperature is correlated with reduced gas pressure within the signal assembly. This ejection of the parachute anchor comprises a catastrophic system failure because when inserted into the full-up system hardware and launched by the rocket motor, the signal assembly would no longer be suspended by a parachute and simply fall to the ground before burnout. In light of this problem, some alternate ignition train designs were considered.

Figure 3. Candle image, postburn; note absence of top protector disc and parachute anchor.

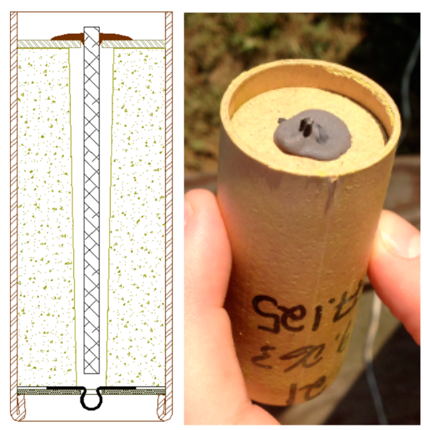
With the problems encountered with formulation **A** in the baseline configuration, two alternate ignition train configurations were developed and tested. In both new configurations, the top protector disc was redesigned with a central 0.794 cm bore to accommodate ventilation of the gases produced by ignition. Specifically for one of the new configurations, the inner candle bore contains only ignition slurry, while the top of the candle bore has a short length of quickmatch held in place perpendicular to the candle bore by a small dollop of ignition slurry (configuration **I**, Figure 4). In the other new configuration, the inner candle bore contains a single strand of quickmatch within the candle bore held in place by ignition slurry coating the inner wall, while the top of the candle bore also has a small dollop of ignition slurry (configuration **II**, Figure 5).



A B

Figure 4. Depictions of full-sized configuration **I**: (A) Cross-sectional drawing with quickmatch (hashed) and slurry (brown). (B) Image of actual candle in configuration **I**.

The outdoor static burn test results of formulation **A** in the two new ignition train configurations are shown below in Table 5. Ten samples in each configuration burned for approximately 15 s, on average, and with adequate color quality. Most



A B

Figure 5. Depictions of full-sized configuration **II**: (A) Cross-sectional drawing with quickmatch (hashed) and slurry (brown). (B) Image of actual candle in configuration **II**.

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Table 5. Static Burn Test Results in Configurations I and II

configuration	burn time (s)	plug failure rate (%)
I	15.67	0
II	15.28	0

importantly, both ignition configurations proved to be viable solutions to the system problem described above because *none of the samples in either configuration* exhibited ejection of the parachute anchor from the signal assembly (i.e., plug failure rate of 0% for both configurations **I** and **II**).

Figure 6 shows the images of the signal assembly after burnout of the yellow smoke formulation, representative of

Figure 6. Candle images, postburn (configurations **I** and **II**): (A) Plug end with roll-crimped parachute anchor intact. (B) Ignition end with protector disc intact.

both configurations **I** and **II**. Note how the parachute anchor remains roll-crimped to the bottom of the candle (Figure 6A), while the bore protector disc retains an interference with the walls of the ignition end of the tube (Figure 6B). With the survival of the signal assembly hardware during static burn testing, additional dynamic burn testing was critical to assess the viability of configurations **I** and **II** in the actual hand-held signal hardware.

Flight Tests. With acceptable static burn test results for formulation **A** in full-sized signal assembly hardware, some preliminary flight tests in full-sized HHS hardware were performed to gain insight into the performance of formulation **A** during ballistic testing. Accordingly, one signal assembly derived from each of the configurations (baseline, **I**, and **II**) was loaded into the full hand-held signal hardware containing the rocket motor and propellant and then deployed on an outdoor test range. Each signal assembly was examined during midburn via digital camera and then again visually after reaching the ground in order to assess any unusual burning behavior (i.e., plug ejection, burning of cardboard tube).

Table 6 shows the performance of all three configurations during ballistic testing. As expected, the signal derived from a candle in the baseline configuration suffered the same ejection problem as before. However, this signal exhibited another fundamental problem; the candle burst into three fragments upon burning of the expelling charge. Figure 7 below shows how the fragments, no longer suspended from a parachute, simply burn while falling in a parabolic path to the ground.

Table 6. Flight Test Results for Signals with Candles in Configurations Baseline, I, and II

configuration	burn time (s)	notes
baseline	13.84	candle burst
I	24.51	incendiary effects
II	15.62	no issues

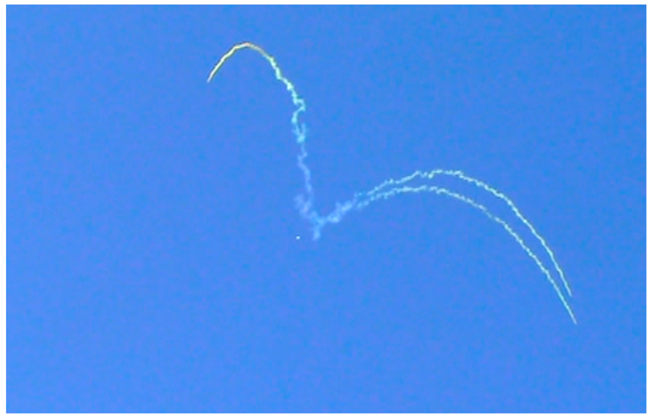

Figure 7. Aerial image of a burning smoke assembly in the baseline configuration.

Another unexpected result ensued from firing the signal derived from a candle in configuration **I**. This time, the payload burned outside the burn time range at about 24 s, and the resulting stream of smoke was remarkably thin. In addition, substantial incendiary effects arose at midburn despite the survival of the parachute and associated mount (Figure 8).

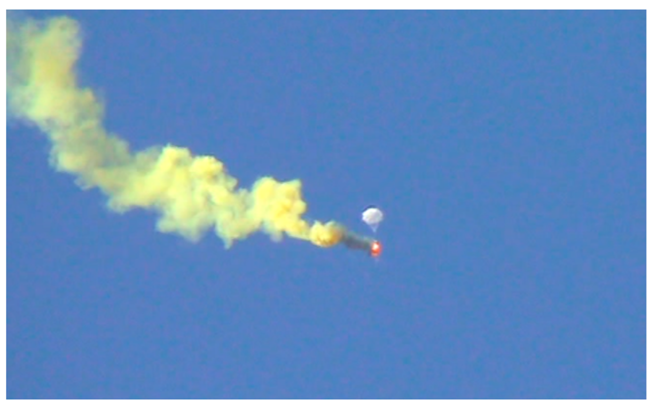

Figure 8. Aerial image of a burning smoke assembly in configuration **I**.

Retrieval of the assembly upon burnout and reaching the ground showed that the cardboard tube had singed considerably. This may be attributed to excessive ignition composition applied to the candle that may have transferred to the tube during flight. While additional flight tests will be needed for further evaluation, these initial results may pose an additional manufacturing challenge for configuration **I**.

The signal loaded with a candle in configuration **II**, however, gave excellent results with formulation **A**, providing a dense yellow smoke cloud well within the required time range. Figure 9 below shows how the parachute is still visibly attached to the signal assembly at midburn. Upon burnout of the yellow smoke payload, the parachute floats to the ground after about a minute, still attached to an empty cardboard tube. In addition,

Figure 9. Image of a burning smoke assembly in configuration **II**.

no undesirable incendiary effects were observed during the aerial burning of the signal assembly in this configuration.

CONCLUSION

In summary, we have advanced a yellow smoke formulation, targeted for insertion into the M194 hand-held signal, from pilot plant operations to a production run. Some mechanical properties of the mixture were assessed, and minor system hardware problems were addressed. Particularly noteworthy are the two novel ignition train configurations that have been demonstrated here in the hand-held signal but may also be extended to many other colored smoke form factors. Furthermore, configuration **II** proved to be the best ignition train configuration for this form factor as it passed static ignition tests and the flight test.

EXPERIMENTAL SECTION

Materials. Potassium chlorate (MIL-P-150D, grade B, Class 7) and sugar (MIL-AA-20135D, Type 1, Style C) were purchased from Hummel Croton, Inc., along with a technical grade preparation of stearic acid. Solvent Yellow 33 (MIL-DTL-51485B(EA), Type II) was purchased from Nation Ford Chemical, Inc. Hydrated basic magnesium carbonate was obtained from Pine Bluff Arsenal (Pine Bluff, AR). All of the pyrotechnic candles were encased in uncoated kraft cardboard tubes.

Preparation of Yellow Smoke Formulations. Granulation studies were performed by adding a 100 g batch of formulation **A** to the top of a vertical stack of sieves, arranged in descending order of pore size and mounted onto a Rotap sieve shaker. The stack was then mechanically agitated for 5 min, and the fractions retained on each sieve were weighed. The retention percentages reported above in Table 2 reflect the averages obtained from testing three 100 g batches of formulation **A**.

Miniature pellets for compressive stress testing were prepared by pressing 3 g of each formulation into a cylindrical geometry (diameter = 1.27 cm, height = 1.45 cm) at 4545.4 kg of dead load, four second dwell. The pellets were crush-tested using an Instron Series 5584 Testing Machine (Instron: Norwood, MA) equipped with a 50 kN load cell. Once each test was started, the load cell was lowered at a rate of 2.54 cm/min until the end of each trial. The end of each trial was indicated by the sensitivity of the load cell, measured here as 40% of the rate of load from the beginning of each trial. The results presented in Table 3 represent the average values obtained from testing 20 pellets derived from formulation **A**.

Formulation A was prepared via a previously reported dry-blending procedure. Full-sized 70 g candles derived from formulation **A** were pressed into noncoated kraft cardboard tubes by our previously reported loading operation or slight modifications thereof.[15] The resulting pyrotechnic candles contained 63.9−72.7 g of energetic material and were coated with a thermate-based ignition slurry consisting of 33.0 wt % potassium nitrate, 24.5 wt % silicon, 20.8 wt % black iron oxide, 12.3 wt % aluminum, 3.8 wt % charcoal, and 5.6 wt % nitrocellulose in acetone. Quickmatch and ignition slurry[15−17] were applied as described in the Results and Discussion section for all three configurations (baseline, **I**, and **II**).

Full-up hand-held signals were loaded with one candle, pressed in the same manner described above and previously.[15−17] Burn times reported above reflect values measured from only one full hand-held signal derived from each of the ignition configurations (baseline, **I**, and **II**).

Characterization. Candles were remotely ignited with an electric match for static ignition tests or lit by the pyrotechnic train of the full hand-held signal during flight tests. Static ignition test data reflect averages from testing 10 candles in each configuration (baseline, **I**, and **II**). Flight test results reflect times measured from deployment of one hand-held signal in each of the three ignition trains described in the Results and Discussion section (baseline, **I**, and **II**). Burn times were measured with a digital stopwatch.

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Author Contributions

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

Notes

The authors declare no competing financial interest.

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DEDICATION

Dedicated to Professor Robert Flowers, II (Lehigh University) on the occasion of his 50th birthday.

ABBREVIATIONS

ARDEC = Armament Research, Development, and Engineering Center; EQT = Environmental Quality Technology; HHS = hand-held signal; OEP = Ordnance Environmental Program; RDECOM = Research, Development, and Engineering Command; U.S. = United States

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